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# Effect of antioxidants on the efficiency of jet milling and the powder characteristics of $Sm_2Co_{17}$ permanent magnets

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This study investigated the effect of antioxidants on the grinding efficiency, magnetic powder characteristics, microstructure, and magnetic properties of 2:17 type SmCo permanent magnet materials. The results show that adding antioxidants helps improve the dispersion among magnetic powders, leading to a 33.3% decrease in jet milling time and a 15.8% increase in magnet powder production yield. Additionally, adding antioxidants enhances the oxidation resistance of the magnetic powders. After being stored in a constant temperature air environment at 25 °C for 48 h, the O content in the powder decreased by 33% compared to samples without antioxidants. While in the magnet body, the O content decreased from 0.21 wt.% to 0.14 wt.%, which helps increase the effective Sm content and domain wall pinning uniformity in the magnet. Excellent magnetic properties were obtained in the magnet with added antioxidants: $B_r = 11.6$ kGs, SF = 79.6%, $H_{cj} = 16.8$ kOe, and $(BH)_{max} = 32.5$ MGOe.

**Keywords:** antioxidant, SmCo permanent magnet, oxidation resistance, grinding efficiency

**PACS:** 81.20.Ev, 75.50.Ww, 81.16.Pr, 81.40.–z **DOI:** 10.1088/1674-1056/ad5537

## 1. Introduction

Due to its high Curie temperature, high coercivity, and relatively high magnetic energy product, 2:17 type SmCo permanent magnet material is widely used in aerospace, communication, and rail transportation fields.[1–5] The 2:17 type SmCo permanent magnet material is generally prepared using a powder metallurgy process with an optimal magnetic performance corresponding to an average particle size of 4–6 μm. When the particle size of the powder is large, there are more internal magnetic domains that hinder orientation and lead to a decrease in residual magnetization. On the other hand, when the particle size is small, although fewer magnetic domains can be obtained, it significantly increases the surface activity of the powder. To reduce its energy level, the powder reacts with O element resulting in an increased proportion of the $Sm_2O_3$ phase in the magnetic material. This further exacerbates the dilution effect on magnetism and deteriorates the performance of magnets.[6–8]

To achieve higher magnetic performance, it is necessary to prepare magnetic powders with smaller particle sizes. However, the oxidation of these powders significantly hampers their application in 2:17 type SmCo sintered permanent magnets. To tackle this issue, researchers commonly resort to operating the magnetic powders within a vacuum glove box, which limits the efficiency of SmCo magnet production and impedes large-scale preparation. Alternatively, surface modification of the powders using additives is another frequently employed approach. The surface of $NdFe_2N_x$(1–4) magnetic powder was modified using a silane coupling agent (KH550), and it was observed that KH550 significantly enhanced the oxidation resistance of $NdFe_2N_x$(1–4) powder.[9] Chen *et al.*[10] incorporated 1 wt.% and 2 wt.% antioxidants during the jet milling and orientation forming stages in the fabrication process of (Nd, MM)–Fe–B (MM represents mixed rare earth) sintered magnets, resulting in effective control over the O content in the magnets. However, there is little research on the application of antioxidants in the pulverizing process of 2:17 SmCo materials, and their influence on the magnetic powder properties of SmCo materials is still unclear.

In this paper, antioxidants were introduced into the preparation process of 2:17 type SmCo sintered magnets to systematically investigate their effects on the efficiency of jet milling and the properties of magnet powder. This work has a practical significance for developing high-energy product magnets and mass-producing high-stability 2:17 type SmCo materials.

## 2. Experimental details

A SmCo alloy ingot with a nominal composition of $Sm(Co_{0.66}Fe_{0.26}Cu_{0.06}Zr_{0.02})_{7.5}$ was prepared using the induction melting method and then crushed into coarse powders

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with a particle size smaller than 300 μm using a medium crusher. An antioxidant mixture solution was obtained by mixing YSH-01 type antioxidant produced by Tianjin Yuecheng New Material Research Institute and 120# solvent oil in a mass ratio of 0.6 wt.% and 0.4 wt.% respectively. The YSH-01 type antioxidant is a medium volatile liquid composed of low molecular polymer with a density of 1.13–1.15 g/ml. The rapid volatilization can be facilitated by heating it at 200 °C. The antioxidant mixture solution was added to the 2 kg coarse powder. It was then mixed in a three-dimensional mixer for 1 h and ground in a fluidized bed jet mill for 6 h, resulting in fine particles with an average particle size ranging from 3 μm to 3.5 μm. The powders were aligned in a magnetic field of 2 T. After compacted by cold isostatic pressing, the green compacts were sintered at 1209 °C for 2 h, solution treatment at 1190 °C for 3 h, followed aged at 840 °C for 12 h, and slow cooled to 400 °C with a cooling rate of 0.7 °C/min and further aged at 400 °C for 3 h.

Particle sizes of the powders were measured by HELOS particle size analysis. The concentration of O element was tested by the oxygen–nitrogen–hydrogen analyzer (ONH836, LECO company, USA). For microstructure observation, samples were prepared by a conventional mechanical grinding and polishing procedure. Microstructure was characterized by scanning electron microscope (SEM, G300, Zeiss company, Germany, operating at 15 kV). The magnetic properties were tested by a *B–H* tracer (NIM-6500C, China). Samples of magnetic properties tests are cylinders with a size of $\Phi 10 \times 10$ mm$^3$. In addition, the microstructure within the grains was detected by transmission electron microscope (TEM, Talos F200x, Thermo Fisher company, USA, operating at 200 kV) equipped with energy dispersive x-ray spectroscopy (EDX).

## 3. Results and discussion

### 3.1. Effect of antioxidant on the efficiency of jet milling

The relationship between the magnetic powder production yield and milling time in the jet milling process is illustrated in Fig. 1 for samples with added and unadded antioxidants. Within the first 0.5 h, the production yield of magnetic powder with added antioxidants was the same as that of magnetic powder without added antioxidants. However, beyond this time point, incorporating antioxidants leads to a significant increase in magnetic powder production yield compared to without added antioxidants. Specifically, after 2 h of jet milling, the magnetic powder production yield rose from 29.5% of unadded antioxidants to 53.9% of added antioxidants. After 4 h of jet milling, the curve representing magnetic powder production yield with added antioxidants tends to plateau, indicating completion of powder production and achieving a production yield as high as 63.2%. However, the production yield of magnetic powder without added antioxidants is only 43%. After 6 h, the jet milling powder without added antioxidants was completed, and the yield was only 54.6%. Compared to magnetic powder without antioxidants added, the jet milling time of magnetic powder with antioxidants added was reduced by 33.3%, while the overall production yield increased by 15.8%. This suggests that fine particles with the addition of antioxidants exhibit higher grinding efficiency and magnetic powder production yield during the jet milling process compared to those without any antioxidants. This holds immense significance in terms of boosting productivity and cutting down on production expenses.

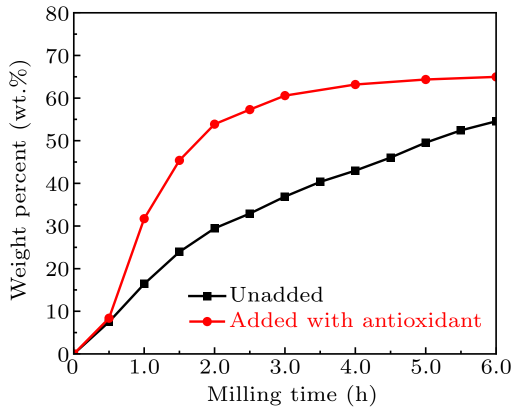


**Fig. 1.** Relationship image of magnetic powder production yield and jet milling time.

### 3.2. Effect of antioxidant on the characteristics of powder

In the jet milling process, the magnetic powder properties also impact the jet milling process, in addition to the parameters of the jet mill. Figure 2 shows the particle size distribution curves of jet milling magnetic powder of added and unadded antioxidants, measured by HELOS particle size analysis. The specific particle size parameters of the two groups of magnetic powder are shown in Table 1. According to the cumulative distribution curve of the two groups of magnetic powders in Fig. 2, it can be found that the magnetic powder with added antioxidants has a higher proportion of small particle size and a lower proportion of large particle size compared to the magnetic powder without added antioxidants. This resulted in a decrease in the surface mean particle size (SMD) of the magnetic particles from 3.49 μm without the addition of antioxidants to 3.12 μm with the addition of antioxidants. According to the particle size distribution parameters of the two groups of magnetic powders in Table 1, it can be found that $D_{10}$ and $D_{90}$ (particle size at 10% and 90% cumulative distribution) of jet milling magnetic powders were 1.86 μm and 9.26 μm, respectively, when no antioxidants were added, and decreased to 1.65 μm and 8.59 μm, respectively, when antioxidants were added.

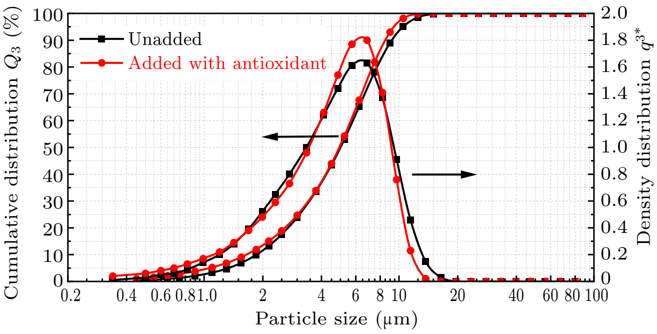


**Fig. 2.** Particle size distribution curves of jet milling magnetic powders of added and unadded antioxidants.

With the decrease in magnetic powder particle size, its surface activation energy increases sharply. To reduce its energy, the magnetic powder will spontaneously gather into clusters, which is called the agglomeration phenomenon.[11] The agglomeration phenomenon among magnetic powders can reduce their dispersion, leading to a decrease in the proportion of small particle sizes that exist separately in the magnetic powder. The decrease in $D_{10}$ and $D_{90}$ indicates an increase in the proportion of small particle size particles present separately in the magnetic powder after the addition of antioxidants. This indicates that adding antioxidants can significantly improve the dispersion between magnetic powders.

**Table 2.** Specific parameters for the magnetic powder.

| Sample | SMD (μm) | $D_{10}$ (μm) | $D_{50}$ (μm) | $D_{90}$ (μm) |
|---|---|---|---|---|
| Unadded | 3.49 | 1.86 | 5.02 | 9.26 |
| Added with antioxidant | 3.12 | 1.65 | 4.94 | 8.59 |

The microscopic morphology of the magnetic powder of added and unadded antioxidants is examined by SEM, as shown in Fig. 3. Both magnetic powders exhibit relatively regular shapes with flat cross sections. Figure 3(a) shows that the magnetic powder without added antioxidants has a more rounded shape, while Fig. 3(b) reveals sharp edges on the surface of the magnetic powder with antioxidants addition. This may happen because the magnetic powder without added antioxidants is not only broken by impact during the jet milling process but also experiences significant wear after crushing, resulting in the loss of surface edges and the formation of rounded particles. In contrast, magnetic powder with antioxidants mainly undergoes treatment through impact fragmentation and is collected before sufficient time for extensive wear, retaining a large number of sharp edges.[12]

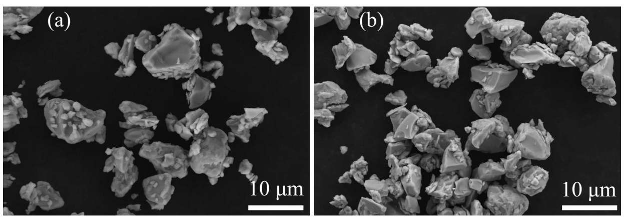


**Fig. 3.** Morphology of magnetic powders with different antioxidant content: (a) unadded, (b) added with antioxidants.

A large number of small particle magnetic powders are attached to the surface of large particle magnetic powders in Fig. 3(a), while the number of small particle particles attached to the surface of large particles in Fig. 3(b) is significantly less. This is consistent with the conclusion shown in Fig. 2 and Table 1 that the dispersion between magnetic particles is improved after the addition of antioxidants.

In addition to the parameters of the jet mill and the feed rate, the grinding efficiency and production yield of the magnetic powder are also influenced by the physical and chemical properties of the magnetic powder itself. For instance, the strength, fluidity, and dispersion of the magnetic powder particles themselves affect the jet milling process, leading to significant variations in magnetic powder obtained through jet milling. During the jet milling process, magnetic powder particles collide with each other and undergo impact fragmentation in the grinding chamber, resulting in surfaces with sharp edges. Particles that were fractured by these impacts experience wear, causing their sharp surface edges to break off into small fragments.[13] However, antioxidants improve the dispersibility and flowability between powder particles.[14] On the one hand, this increases the collision rate and wear rate among magnetic powders in the grinding chamber. On the other hand, it allows those magnetic powders that meet collection requirements but cannot pass through the sorting wheel due to agglomeration to be collected more quickly.[15] This significantly reduces the time required for jet milling and improves efficiency in preparing magnetic powders using this method (as shown in Fig. 1).[15–17] However, since they are rapidly collected within the chamber without a sufficient degree of wear on their surfaces, a large number of sharp edges remain on these collected magnetic powders (as shown in Fig. 3(b)).

To investigate the impact of antioxidants on the oxidation resistance of the magnetic powder, both jet milling magnetic powder of added and unadded antioxidants were separately placed in a constant temperature drying cabinet. The O content of each magnetic powder was measured at different time points, and a curve depicting the change in O content over time was obtained, as shown in Fig. 4. At the initial stage, there is a significant difference in O content between the two magnetic powders, the magnetic powder without added antioxidants has a significantly higher O content compared to the magnetic powder with the addition of antioxidants. This difference is caused by the contact between the magnetic powder and O element during both the preparation stage of the magnetic powder and the sample preparation process. As time progressed, both samples initially increased and then leveled off. After 48 h of placement, the magnetic powder with the addition of antioxidants maintained an O content of 0.32 wt.%, which was 33% lower than that in the magnetic powder without added antioxidants. The results indicate that the antioxidation capacity of the magnetic powder is significantly higher than that of the magnetic powder without an antioxidant. The presence of the antioxidant coating on the surface of the material particles reduces the extent of the reaction between the magnetic powder and O elements, thereby improving its oxidation resistance.

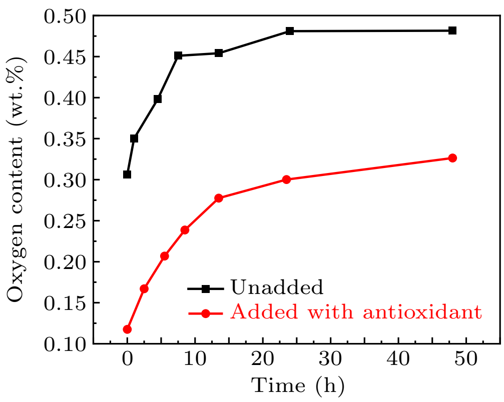


**Fig. 4.** Relationship between O content of magnetic powder and time.

### 3.3. Effect of antioxidant on the microstructure and magnetic properties

Backscatter electron (BSE) images for the magnets of added and unadded antioxidants as shown in Figs. 5(a) and 5(b). In these two groups of magnets, three phases of different contrast can be observed: a gray matrix phase, a white $Sm_2O_3$ phase, and a dark gray Zr-rich phase.[18–23] The O element mainly exists in the form of the $Sm_2O_3$ phase in $Sm_2Co_{17}$ permanent magnets.[7,24] Excessive O element will reduce the effective Sm content and functional phase in the magnet, thereby deteriorating its structure and magnetic properties.[25] In the magnet without added antioxidants, a significant amount of white $Sm_2O_3$ phase is present. However, in the magnet with added antioxidants, the quantity and volume of the $Sm_2O_3$ phase are noticeably reduced. Figure 5(c) shows the O content in the corresponding magnets, revealing that compared to magnets without added antioxidants, those with added antioxidants exhibit a decrease in O content from 0.21 wt.% to 0.14 wt.%. After adding antioxidants, the magnetic powder's oxidation resistance is enhanced, which reduces the O content adsorbed in the magnetic powder, and the O content in the magnet is also reduced. Studies have shown that for each unit mass of O element, 6.27 times the Sm element needs to be lost to combine with it to form the $Sm_2O_3$ phase.[26] According to the calculation, the Sm loss in the magnet without added antioxidants was 1.32 wt.%, while in the magnet with added antioxidants, the Sm loss was 0.88 wt.%. Therefore, magnets with added antioxidants have a higher effective Sm content. In addition, a large number of black holes will be produced in the magnets with added antioxidants, as indicated by the red arrow in Fig. 5(b). The YSH-01 type antioxidant evaporates rapidly when heated at temperatures below 200 °C. Therefore, the holes in the magnets may be caused by the volatilization of antioxidants during heat treatment. The increase of holes reduces the magnet density from 8.36 g/cm$^3$ without adding antioxidants to 8.31 g/cm$^3$ with adding antioxidants, as shown in Fig. 5(d).

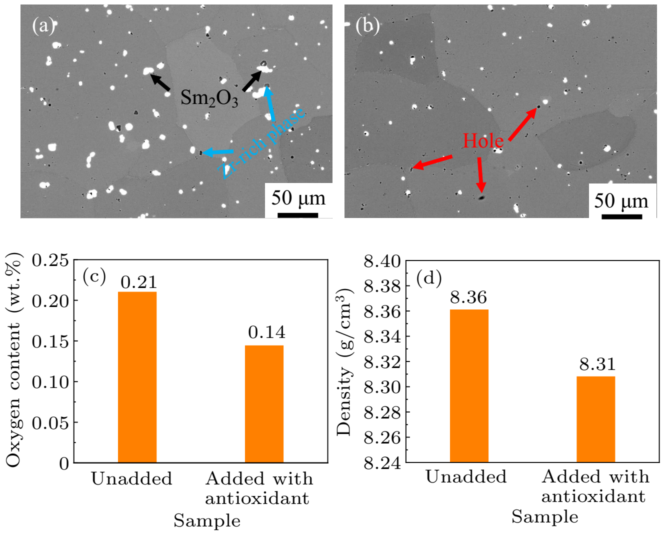


**Fig. 5.** BSE images of magnets: (a) unadded, (b) added with antioxidant, (c) O content in magnets of added and unadded antioxidants, (d) magnets density of added and unadded antioxidants.

Figure 6 shows TEM bright field images of added and unadded antioxidant magnets, high-resolution images of cell wall interaction, and statistical diagrams of cell structure size. A complete cellular structure can be formed in both groups of magnets, and the average size of the cellular structure in the two groups of magnets is the same. However, the thickness of the cell wall phase increased from 9.15 nm without the addition of antioxidants to 13.44 nm with the addition of antioxidants. This may be caused by the increased effective Sm content in the magnets with added antioxidants. When the average size of the cellular structure is unchanged, the increase in the thickness of the cell wall phase means an increase in the volume of the cell wall phase in the magnet, which will lead to a decrease in the concentration of Cu in the cell wall phase.[27]

The demagnetization curves of the magnet of added and unadded antioxidants are shown in Fig. 7. Based on the specific magnetic performance parameters in Table 2, the following changes can be observed: compared to the magnet without added antioxidant, the magnet with added antioxidant exhibits an increase in remanence ($B_r$) from 11.5 kGs to 11.6 kGs, an increase in squareness factor (SF) from 71.3% to 79.6%, and an improvement in maximum magnetic energy product from 32.3 MGOe to 32.5 MGOe. Only coercivity ($H_{cj}$) shows a decrease from 20.8 kOe to 16.8 kOe. The SF is represented by $H_k/H_{cj}$, where $H_k$ is the field at which the demagnetization curve reaches its inflection point and Jr retains 80%.[18]

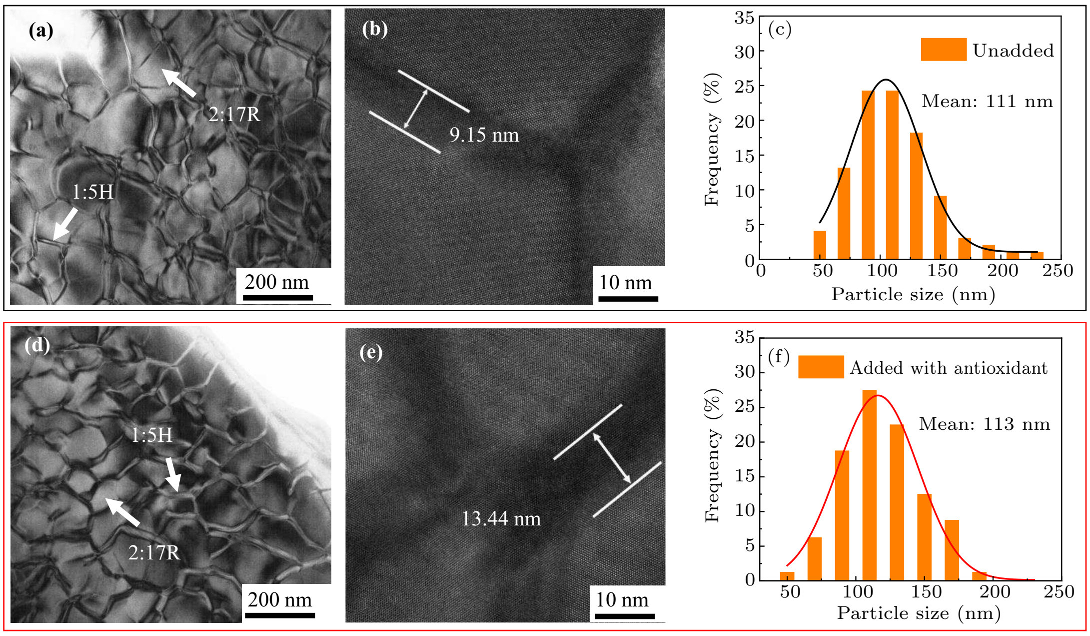


**Fig. 6.** TEM bright field image, HRTEM images of the cell wall phase, and cell structure size distribution histogram of aged magnet grain for two groups of magnets: (a)–(c) unadded, (d)–(f) added with antioxidant.

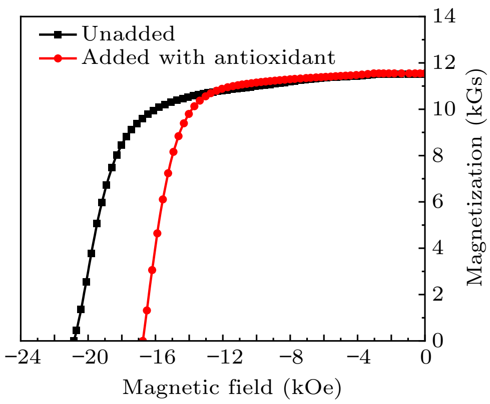


**Fig. 7.** Demagnetization curve of magnets.

The remanence of anisotropic sintered magnets can be expressed as

$$B_r = A\overline{\cos\theta}(1-\beta)\rho\mu_0 M_s \tag{1}$$

with $A$ the exchange constant, $\overline{\cos\theta}$ the orientation factor, $\theta$ the angle between the orientation axis of the magnet and easy magnetic axis in the grain, $\beta$ the volume percentage of the nonmagnetic phase, $\rho$ the density, $\mu_0$ the permeability of vacuum, and $M_s$ the magnetic saturation magnetization.[28] The $Sm_2O_3$ is the nonmagnetic phase, and its decrease contributes to the improvement of remanence. However, the increase in the number of holes in magnets with added antioxidants will reduce the magnet's density, leading to remanence deterioration. The combined effect of the two increases the remanence of the magnet with added antioxidants, but the increase is relatively small. In addition, studies have shown that magnetization reversal initially occurs at the ferromagnetic grain boundaries or in the vicinity of nonmagnetic Sm oxides. As the demagnetization field increases after magnetization reversal, the reversal region extends into the grain from these areas by the magnetic domain wall motion.[29] After the addition of antioxidants, the $Sm_2O_3$ phase in the magnet decreases, the preferential demagnetization region decreases, and the SF of the magnet increases. After the addition of antioxidants, the effective rare earth content in the magnet increases, increasing the cell wall phase volume fraction, the decrease of Cu concentration in the cell wall phase, and the decrease of coercivity of the magnet. In addition, this paper only focuses on the effects of antioxidants on $Sm_2Co_{17}$ powder properties, microstructure, and magnetic properties. These effects contain both positive and negative aspects. Eliminating the adverse effects through the appropriate technology still needs further research.

**Table 2.** Particle size distribution curves of jet milling magnetic powders of added and unadded antioxidants.

| Sample | $B_r$ (kGs) | $H_{cj}$ (kOe) | $(BH)_{max}$ (MGOe) | SF (%) |
|---|---|---|---|---|
| Unadded | 11.5 | 20.8 | 32.3 | 71.3 |
| Added with antioxidant | 11.6 | 16.8 | 32.5 | 79.6 |

## 4. Conclusion

This paper has systematically studied the effects of antioxidants on jet milling efficiency, magnetic powder characteristics, magnet microstructure, and magnetic properties. The following conclusions can be drawn.

1. By adding antioxidants to the magnetic powder, its dispersibility is enhanced, thereby increasing its collision frequency in the grinding chamber. This resulted in the jet milling time being reduced by 33.3% and the final production yield increased by 15.8%.

2. After jet milling, the magnetic powder doped with antioxidants was kept at 25 °C for 48 h. It was found that the O content of the powder decreased by 33% compared to the un-

doped magnetic powder, indicating a significant improvement in its antioxidant properties.

3. By adding antioxidants, the magnet's remanence and SF can be improved by decreasing the number of nonmagnetic $Sm_2O_3$ phases. The density of magnets with added antioxidants will be reduced by the increase in holes, which will limit the improvement of remanence.

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